

Defect annealing studies on metals by positron annihilation and electrical resistivity measurements

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Doppler-broadening positron-annihilation measurements combined with electrical-resistivity measurements were performed on Cu and Al samples, irradiated by high-energy electrons at liquid-helium temperature. A defect-specific parameter R was determined from the shape of the 511-keV annihilation line. The R parameter is found to be independent of the defect concentration C and the positron trapping constant μ within the framework of the two-state trapping model. During isochronal annealing of electron-irradiated copper the line-shape parameter ΔI , increases in stage III, whereas the electrical resistivity decreases, indicating a reduction in the Frenkel-defect concentration. These two combined effects and the pronounced steps in the R parameter during stage-III annealing, which are found to be dose independent, signify that radiation-produced vacancies become mobile and coalesce into clusters before they anneal out. The positron trapping constant μ for vacancies in Cu was determined to be $\mu = (4.25 \pm 0.8) \times 10^{14} \text{ sec}^{-1}$. Furthermore, the annealing behavior of copper and aluminum samples, deformed plastically at 77 K and irradiated at 4.2 K by electrons, respectively, is discussed. In both cases no indication for the formation of vacancy agglomerates is found during annealing. The R -parameter analysis is also applied to the data for electron-irradiated Mo, as reported by Eldrup *et al.*

I. INTRODUCTION

In order to study the annealing behavior of defects, one requires experimental techniques which provide specific information about the type of defects involved. The methods applied so far, e.g., electrical resistivity,¹ electron microscopy,² high-voltage electron microscopy,³ diffuse x-ray scattering,⁴ etc., are either not selective as to the type of defect present in the sample or respond mainly to single interstitials and/or to extended defect agglomerates. The positron-annihilation technique can be used as a method in defect-recovery studies, which is mainly sensitive to vacancy-type defects. The high sensitivity of positrons to vacancies has frequently been demonstrated in thermal-equilibrium measurements, where accurate vacancy-formation enthalpies could be obtained.^{5,6}

The primary aim of this work was to elucidate some of the open problems in defect-annealing behavior and to show that positron-annihilation techniques can lead to defect-specific quantities. It makes this method a powerful tool in defect-recovery studies and complementary to other techniques. Particular emphasis was placed on the annealing behavior of electron-irradiated and cold-worked Cu. Furthermore, the annealing of electron-irradiated Al and Mo is discussed. The annihilation characteristics of the positron-electron pair into two 511-keV photons depend on the mo-

mentum distribution of the electrons and on the state of the positron.⁷ The positron is in a free Bloch state in a perfect crystal and in a bound state, when trapped by a crystal-lattice defect, e.g., a vacancy. Positrons can be trapped not only by vacancy-type defects, e.g., vacancies, vacancy agglomerates, and voids, but also by dislocations and dislocation loops.⁸

However, there are several ways in positron-annihilation experiments to identify the type of defect present in the sample: High-precision lifetime measurements provide the individual positron lifetimes in the free and trapped states,⁹ where the lifetime of the positrons in the trapped states characterizes the kind of defect. However, the individual lifetimes are very short ($\sim 10^{-10}$ sec) and they can be resolved only by very refined techniques and sophisticated computer analysis, complicating the interpretation of the results.

Angular-correlation measurements of positrons annihilating from vacancies in thermal equilibrium and from voids in a metal show marked differences in the photon-momentum distribution.¹⁰ This behavior implies that the shape of the angular-correlation curve or the shape of the Doppler-broadened spectrum of the 511-keV annihilation radiation depends sensitively on the type of defect-trapping positrons in the metal. Under these circumstances a more-detailed analysis of the shape of the measured curves seems to be necessary. One possibility, proposed by Eldrup *et al.*,¹¹ is

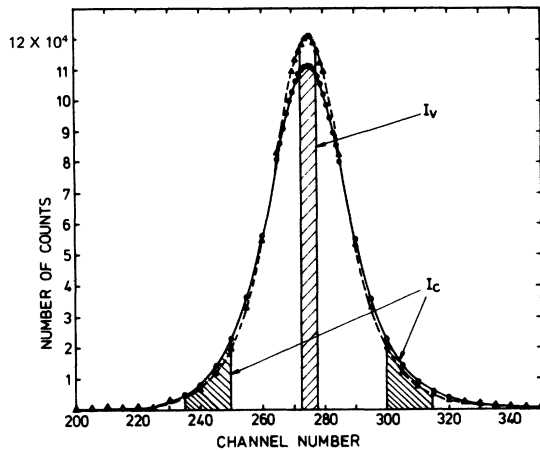


FIG. 1. Doppler-broadened 511-keV annihilation line shapes of annealed (circles) and of electron-irradiated (triangles) Cu with the line-shape parameters I_v and I_c indicated.

fitting the angular-correlation curves by a sum of Gaussian functions, where the contributions due to annihilations in the perfect lattice and from each defect type is represented by one, two, or three Gaussians (and/or parabolas), respectively. This procedure (called PAAC FIT) demands a complete series of measurements in order to evaluate the characteristic parameters by a fitting procedure—the full widths at half-maximum and the relative intensities—of the Gaussians (parabolas) for each defect type. The final analysis provides useful information not only concerning the type of the defect, but also about the relative number of positrons trapped by these defects.

Based on the trapping model, we propose an analysis of the 511-keV radiation line shape which leads to a defect-specific parameter R ,^{12, 13} and could make positron-annihilation measurements a standard tool in materials research. The analysis is applicable for Doppler broadening as well as for angular-correlation measurements.

For the calculation of the R parameter we use the two line-shape parameters I_v and I_c (Fig. 1). I_v is a small portion in the center of the Doppler-broadened 511-keV spectrum divided by the total area. The width of this central portion is equivalent to about 2 mrad in angular-correlation spectra. The definition of I_v is guided by the definition of the quantity measured by coincidence peak counts in angular-correlation experiments. I_c is the sum of two segments on either side of the peak (corresponding to the region from about 10–20 mrad). Thus, I_v is a measure of annihilations of positrons with conduction and core electrons, and I_c is associated with core electrons alone. If I_v^f and I_v^t are the characteristic values of the line-

shape parameter I_v in the free and trapped states, respectively, then we have

$$I_v = I_v^f P_f + I_v^t P_t, \quad (1)$$

where P_f and P_t are the relative probabilities ($P_f + P_t = 1$). P_t is given by

$$P_t = \mu C / (\lambda_f + \mu C), \quad (2)$$

where μ is the trapping constant, C is the trap concentration, and λ_f is the annihilation rate from the free state. If we substitute Eq. (2) into Eq. (1), we find

$$I_v = I_v^f \left(1 - \frac{\mu C}{\lambda_f + \mu C}\right) + I_v^t \frac{\mu C}{\lambda_f + \mu C}. \quad (3)$$

An equivalent relation can be deduced for the line-shape parameter I_c . Then the changes in the two parameters due to trapping can be written

$$I_v - I_v^f = (I_v^t - I_v^f) \mu C / (\lambda_f + \mu C), \quad (4)$$

$$I_c - I_c^f = (I_c^t - I_c^f) \mu C / (\lambda_f + \mu C). \quad (5)$$

These differences still depend on the defect concentration. A characteristic parameter R is obtained by taking the ratio defined previously,¹² namely,

$$R = \left| \frac{I_v - I_v^f}{I_c - I_c^f} \right| = \left| \frac{I_v^t - I_v^f}{I_c^t - I_c^f} \right|. \quad (6)$$

Here, R is concentration independent and characterizes the type of trapping site involved. This is valid if only one type of trapping center is present. If several types of trapping sites are available for the positrons at the same time, the above argument is also valid as long as one type predominates.

II. EXPERIMENTAL PROCEDURE

We used polycrystalline samples of dimensions 10×12 mm, prepared from 99.999% stated-purity copper and from 99.999% stated-purity aluminum. The thickness was about 0.3 mm for Cu and about 0.45 mm for Al. Another sample with the same thickness and of dimensions 20×0.5 mm, prepared identically, was used for resistivity measurements. The specimens for the resistivity measurements had residual-resistivity ratios $\rho(300 \text{ K}) / \rho(4.2 \text{ K})$ of 1000–1500 in the case of copper and of around 1500 in the case of aluminum.

The two samples for the positron-annihilation experiments and the sample for the resistivity measurements were irradiated simultaneously with 3-MeV electrons at about liquid-helium temperature at the Jülich low-temperature irradiation facility to different doses. After the irradiation, the positron samples were shifted in such a way that after inserting a 10- μCi ^{22}Na positron source

between the samples, a sandwich arrangement was achieved. The samples were held at 4.2 K in a helium cryostat during the transport into the laboratory.

The Doppler-broadening measurements were performed with a Ge(Li) detector, having an energy resolution of 1.12 keV at 514 keV at a rate of 1000 counts/sec. The electronic equipment was temperature stabilized within a few tenths of a degree. The results on electron-irradiated samples were obtained by the measuring procedure described in Ref. 12. The measurements on cold-worked Cu and neutron-irradiated Al were achieved with a new computer-stabilization technique. We stabilize the Doppler-broadened 511-keV peak by monitoring simultaneously the 497-keV γ line of ^{103}Ru . A PDP 11/40 computer with CAMAC electronics is used as a programmable multi-channel analyzer. The final spectrum consists of a sum of 70–100 spectra, where each spectrum is accumulated for a period of 100 sec only, resulting in a total number of counts of 4×10^6 . These spectra are corrected mathematically for shifts by the electronics before being added together. With a total rate of 10^4 counts/sec, this procedure causes a statistical broadening of the full width at half-maximum (FWHM) of the resolution peak of less than ± 0.04 channel, where the width of one channel corresponds to 103 eV. Such a broadening of about 0.3% is negligible, but the long-time stability is guaranteed due to this stabilization technique. Therefore, this procedure gives a definite advantage, in that the electronic system is precisely monitored, and it also provides the resolution function of the detector, which contains the same statistical shifts as for the 511-keV line.

III. RESULTS AND DISCUSSION

A. R parameter

Figure 2 shows the R parameter versus Frenkel-defect concentration C of electron-irradiated copper. The data points displayed in Fig. 2 were calculated from the line-shape parameters obtained for three different irradiations (see Fig. 4) and successive isochronal annealing below 210 K, where the vacancies are the dominant trapping sites for the positrons. The defect concentrations were determined by electrical-resistivity measurements using the relation $C = \Delta\rho/\rho_{\text{FD}}$, where $\Delta\rho$ denotes the specific resistivity increase due to the defects and $\rho_{\text{FD}} = 2.5 \mu\Omega \text{ cm/at. \%}$ denotes the specific resistivity per Frenkel pair.¹⁴ The R parameter remains constant over the investigated concentration region from 2×10^{-5} to 4×10^{-4} , whereas the individual line-shape parameters change drastically in this region, as will be dis-

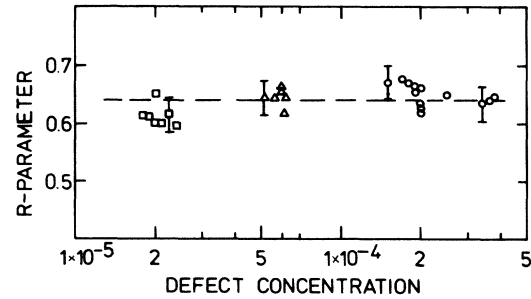


FIG. 2. R parameter vs Frenkel-defect concentrations for electron-irradiated Cu.

cussed later (Fig. 4). This shows, experimentally, that the R parameter is independent of the defect concentration C and of the trapping constant μ .

Figure 3 gives the values of the R parameter for different concentrations and various sizes of voids in neutron-irradiated aluminum versus temperature of the sample. In Ref. 8 it has been shown that the line-shape parameters depend on the void concentrations and on the sizes of the voids as well as on the temperature of the sample, due to the strong temperature dependence of the positron-trapping constant μ .

The constant values of the R parameter in Fig. 3 show that the R parameter does not depend on the concentration C and on the trapping constant μ , but is independent of both. The void densities and the mean diameters of the voids given in Fig. 3 were determined by electron microscopy.⁸ The R value for single vacancies in Al, obtained after electron irradiation (see also Fig. 7), is also given

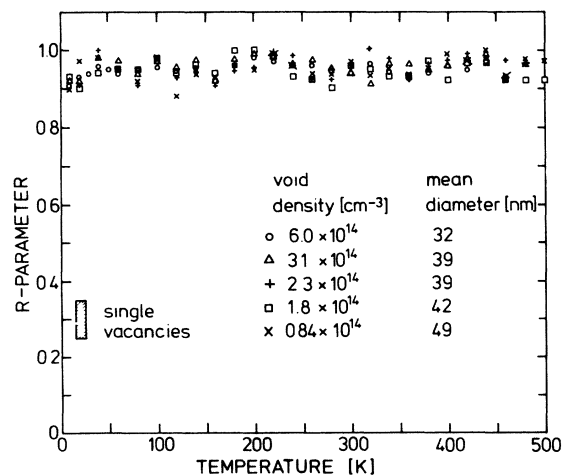


FIG. 3. R parameter for different concentrations and various sizes of voids in neutron-irradiated Al (Ref. 8) vs sample temperature. The R value for single vacancies in Al is also indicated.

in Fig. 3. This behavior makes the R parameter a very helpful defect-specific quantity in annealing studies.

B. Annealing of electron-irradiated copper

Cu samples were irradiated to different doses corresponding to residual-resistivity changes of 126×10^{-9} , 65×10^{-9} , and $30 \times 10^{-9} \Omega \text{ cm}$.¹⁵ For the highest dose the isochronal annealing was started at 31 K. In the case of the highest and the lowest dose, each temperature step was 10% of the preceding temperature, whereas for the medium dose the temperature steps were 15%. All positron-annihilation measurements were performed at 10 K, while the resistivity measurements were done at 4.2 K. The results are shown in Fig. 4, where ΔI_v is the difference between the line-shape parameter I_v and the characteristic value for the annealed specimen I_v^f . In recovery stage I, ΔI_v decreases due to the decrease in the number of vacancies in this stage, i.e., parallel to $\Delta\rho/\Delta\rho_0$.

At lower doses a slight decrease in ΔI_v is also observed in stage II. The most startling effect, however, is the increase of the line-shape-parameter difference ΔI_v in the annealing stage III, where the resistivity decreases drastically. If the positrons were being trapped by the same type of defect as in stages I and II, ΔI_v should decrease too. The increase of ΔI_v in stage III indicates that the type of the dominating trapping sites has

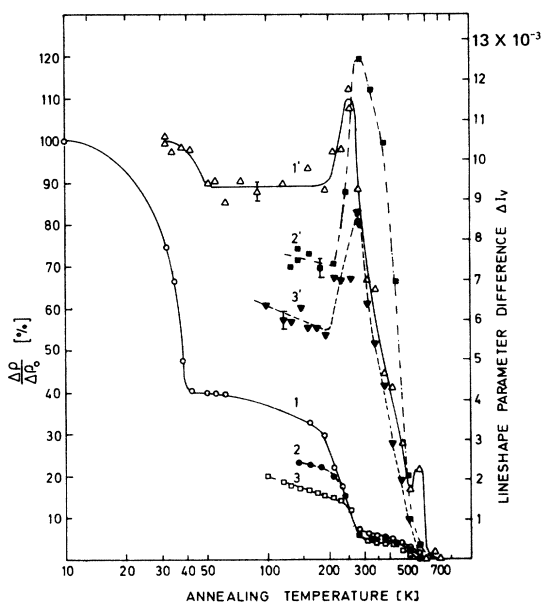


FIG. 4. Line-shape-parameter difference ΔI_v and resistivity $\Delta\rho/\Delta\rho_0$ as a function of the annealing temperature for electron-irradiated Cu for different doses: 1, $\Delta\rho_0 = 126 \text{ n}\Omega \text{ cm}$; 2, $\Delta\rho_0 = 65 \text{ n}\Omega \text{ cm}$; 3, $\Delta\rho_0 = 30 \text{ n}\Omega \text{ cm}$.

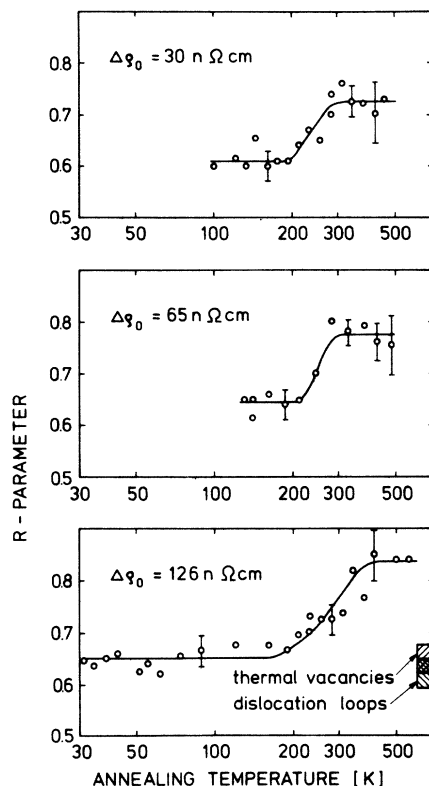


FIG. 5. R parameters vs annealing temperature of electron-irradiated Cu for different doses. The values of R for vacancies in thermal equilibrium and for dislocation loops are indicated in the lower graph.

changed. Further annealing results in a decrease of ΔI_v . The recovery process in copper is completed at about 650 K, when resistivity and line-shape parameters return to the values before the electron irradiation.

Figure 5 shows the changes of the defect-specific R parameter versus annealing temperature for the three different doses. During stages I and II, the R parameter is constant and has a value between 0.61 and 0.65, in spite of the well-known formation of small interstitial clusters during annealing stage II.¹⁶ The rise in R during annealing stage III indicates that the nature of the dominating trapping center is changing independently of the dose. From electron microscopy¹⁷ it is known that interstitials are present in the form of dislocation loops above stage III. However, dislocation loops cannot be the dominant traps because their characteristic R parameter, determined from copper single crystals containing dislocation loops, is 0.62 ± 0.03 , which is too small to explain this step. These dislocation loops were produced by neutron irradiation above room temperature.¹⁸ About the same value for R was also observed for disloca-

tions in cold-worked copper (Fig. 7).

In order to compare R values obtained by angular correlation and by Doppler-broadening measurements, we evaluated the R parameter for dislocation loops in copper single crystals with both methods. The R value measured by angular correlation turned out to be about the same as the value for thermal vacancies obtained also by angular-correlation studies.¹⁹ Since the R parameter for dislocation loops was determined by both methods, we can compare the value for vacancies in thermal equilibrium on the scale for the Doppler results, due to the equivalence of both methods. It is important to mention that the R parameter for single vacancies, given in Fig. 5, is almost identical to the R values obtained for annealing stages I and II. Since interstitial-type defects can be excluded by the above arguments to be responsible for the rise in R , we conclude that radiation-induced vacancies become mobile in annealing stage III and coalesce into small, probably three-dimensional clusters. When these clusters grow larger most of them collapse into vacancy loops, which can be seen in electron-microscopy investigations.¹⁷

C. Trapping constant and formation entropy of vacancies in copper

The combined measurement of the line-shape parameters and the electrical resistivity allows the evaluation of the trapping constant μ , since the electrical resistivity is a measure of the defect concentration. Equation (3), deduced from the trapping model, can also be written

$$(I_v - I_v^f)/(I_v^t - I_v) = (\mu/\lambda_f)C. \quad (7)$$

This relation plotted on a double-logarithmic scale (Fig. 6), gives a straight line with the intercept of $\ln(\mu/\lambda_f)$ on the ordinate and the slope of 1. The

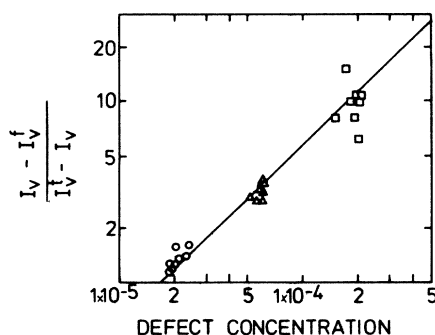


FIG. 6. Double-logarithmic plot of the relation $(I_v - I_v^f)/(I_v^t - I_v) = (\mu/\lambda_f)C$ for electron-irradiated Cu for various Frenkel-defect concentrations. The different symbols (\circ , Δ , \square) correspond to different irradiations.

data points shown in Fig. 6 were obtained from the three samples irradiated at various doses as described above. Here, only the points below annealing stage III were used, where mainly single vacancies act as trapping sites for the positrons. With $\lambda_f = 0.746 \times 10^{10} \text{ sec}^{-1}$,²⁰ we obtain a value of $(4.25 \pm 0.8) \times 10^{14} \text{ sec}^{-1}$ for the trapping constant μ for vacancies in copper.

In Refs. 8 and 21 it has been shown that the trapping constant for vacancies is temperature independent below room temperature. Theoretical arguments²² support the assumption that μ is temperature independent for vacancies. Positron experiments of metals containing vacancies in thermal equilibrium can provide the formation enthalpies H_{1v} of vacancies and the values of the term $\mu e^{S_{1v}/k}$, where S_{1v} denotes the formation entropy and k is the Boltzmann constant. Combining these data from the literature^{6,23-25} with the above value for μ , a value of $S_{1v} = (2.6 \pm 0.5)k$ for Cu is deduced. This value is in agreement with the result of $S_{1v} = 2.35k$ obtained from recent quenching experiments²⁶ combined with the results of Ref. 27. Most recent theoretical calculations²⁸ provide a value of $S_{1v} = (2.05 \pm 0.25)k$.

D. Annealing of plastically deformed copper

The high sensitivity of positrons to vacancy-type defects can also be used to investigate the annealing behavior of plastically deformed copper. Severely cold-worked metals contain point defects and dislocations with concentrations of the order of about 10^{-4} and 10^{11} – 10^{12} cm^{-2} , respectively.²⁹ The interaction of point defects with dislocations, dislocation loops, etc., plays an important role in void formation and irradiation creep.³⁰ Recent measurements of the damage-production rate in copper,³¹ deformed at 78 K, were interpreted by assuming a strong interaction of migrating self-interstitials with small-vacancy and interstitial clusters in addition to the interaction of self-interstitials with dislocations. A positron experiment could reveal the role of vacancy clusters during the annealing of plastically deformed metals.

We deformed pure-Cu specimens at liquid-nitrogen temperature by rolling, and maintained the samples below 100 K during the transfer into the cryostat. The Doppler broadening was measured at 77 K after each isochronal annealing step. The results are shown in Fig. 7. The line-shape parameter ΔI_v shows a monotonous decrease with a pronounced annealing step around 300 K. The annealing is completed at about 700 K. The R parameter remains constant within the statistical accuracy during annealing. Its value is too small (see Fig. 5) to indicate the existence or formation

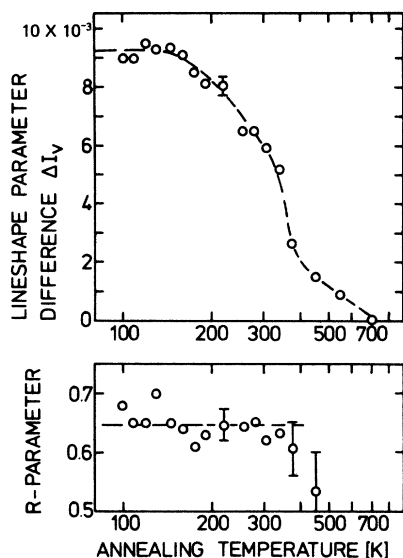


FIG. 7. ΔI_v and R parameter vs annealing temperature for Cu plastically deformed at 78 K.

of vacancy agglomerates. The R value of 0.65 is typical for dislocations and dislocation loops as well as for single vacancies in Cu. This indicates that positron trapping in plastically deformed copper during annealing is dominated by dislocations and interstitial clusters, whereas vacancy agglomerates are either not formed or their concentration is too small to cause an observable effect in the presence of the high-dislocation density.

E. Annealing of electron-irradiated aluminum

The Al specimens were irradiated to a dose corresponding to a residual-resistivity change of $\Delta\rho_0 = 140 \times 10^{-9}$ cm. The isochronal annealing was started at 60 K. Figure 8 shows the annealing behavior of the line-shape-parameter difference ΔI_v , and of the electrical resistivity. The Frenkel-defect concentration at 60 K was about 1.0×10^{-4} , assuming $\rho_{FD} = 4.2 \mu\Omega \text{ cm/at. \%}$.⁴ The slight increase of ΔI_v around 100 K is almost within the statistical uncertainty, but, in principle, it could be due to the formation of interstitial clusters. In contrast to the results for copper, the line-shape parameter does not increase during annealing stage III, but drops in a similar way as the electrical resistivity does.

Figure 8 shows also the annealing behavior of the R parameter. Its value close to 0.3 is representative for vacancies, since vacancies are the dominant trapping sites for positrons after electron irradiation. This value is about three times smaller than obtained for voids in Al, shown in Fig. 3. Due

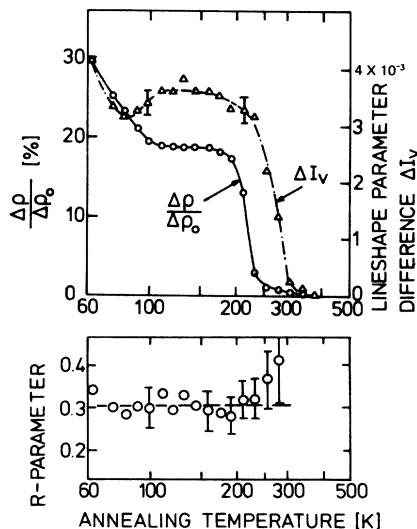


FIG. 8. Line-shape-parameter difference ΔI_v and resistivity $\Delta\rho/\Delta\rho_0$, as well as R parameter annealing temperature for electron-irradiated Al ($\Delta\rho_0 = 140 \text{ m}\Omega \text{ cm}$).

to the rather small trapping effect above 250 K, the slight increase in R in this region must be attributed to statistical fluctuations and gives no evidence for vacancy clustering.

The combined marked decrease in stage III in the line-shape parameter ΔI_v , and in the resistivity $\Delta\rho/\Delta\rho_0$, signifies a decrease in the number of trapping sites as well as in the defect concentration, and indicates that vacancies become mobile and combine with interstitials or interstitial clusters. Huang scattering measurements³² have shown that interstitial agglomerates are formed during stage II, and since the interstitial atoms within the cluster are very strongly bound, only vacancies can migrate at these temperatures. However, no indication is given for the formation of vacancy agglomerates.

F. Electron-irradiated molybdenum

Eldrup *et al.*¹¹ irradiated pure polycrystalline Mo samples with 10-MeV electrons to a dose of 2×10^{18} electrons cm^{-2} at approximately 50 °C. Positron-lifetime and angular-correlation measurements combined with resistivity measurements and electron-microscope studies during isochronal annealing demonstrated clearly, that vacancies become mobile in stage III (150–350 °C), which leads to the formation of small voids in electron-irradiated Mo. The angular-correlation results were analyzed by the above-mentioned PAAC FIT procedure, which gave a pronounced increase in the intensity of the void component

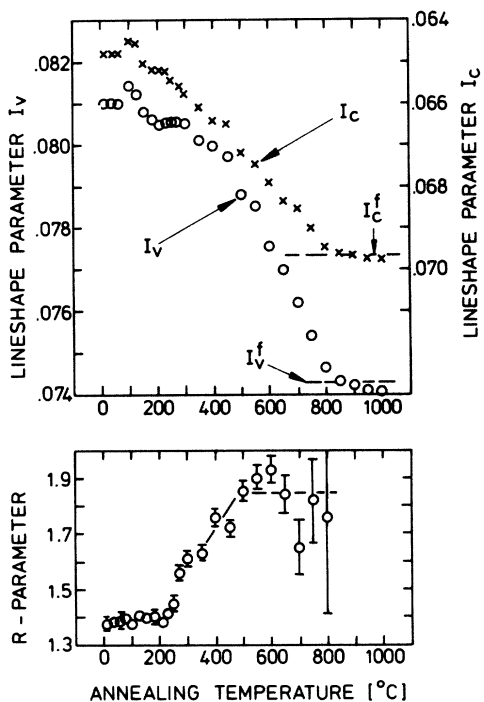


FIG. 9. Line-shape parameter I_v and I_c and R parameter vs annealing temperature for electron-irradiated Mo. The I_c parameter and the R parameter were calculated from the data obtained by Eldrup *et al.* (Ref. 11).

above about 200 °C.

The I_c parameter and the R parameter shown in Fig. 9 were calculated from the data given in Ref. 11. Figure 9 shows the line-shape parameters I_v and I_c as well as the corresponding R parameter. The rise in the R parameter above 200 °C suggests a change in the type of defect-trapping positrons in the sample. The increase of R —similar to the Cu-data—indicates the coalescence of vacancies to small clusters or voids. This is in complete agreement with the results in Ref. 11. Here, it is shown that the R -parameter analysis leads to the same basic conclusions as gained from other much-more-complex procedures.

IV. CONCLUSIONS

Positron-annihilation techniques combined with electrical-resistivity measurements were applied to investigate the annealing behavior of radiation-induced defects in Cu and Al. The recovery of cold-worked copper was also studied by positron annihilation. The determination of a defect-specific parameter R from the shape of the Doppler-broadened curves of the 511-keV line or from angular-correlation curves leads to defect-specific informations. During annealing stage III of electron-irradiated Cu the increase of the line-shape parameter ΔI_v and the drop of the resistivity signify a loss in the defect concentration and an increase in the trap size. This behavior combined with the dose-independent increase of the R parameter indicates the formation of vacancy agglomerates and implies free migration of vacancies in copper in annealing stage III.

The trapping constant μ for vacancies in Cu was determined to be $\mu = (4.25 \pm 0.8) \times 10^{14} \text{ sec}^{-1}$. The formation entropy S_{1v} of vacancies in Cu was calculated from thermal-equilibrium data and the trapping constant μ to be $S_{1v} = (2.6 \pm 0.5)k$, assuming a temperature-independent trapping constant.

Annealing results on plastically deformed Cu show a marked annealing step around room temperature but do not indicate any formation of vacancy agglomerates. In electron-irradiated Al complete annealing in resistivity and line-shape parameters around 300 K is achieved. Vacancy agglomerates are not observed. In the case of electron-irradiated Mo the R parameter analysis lent further support to the results as obtained by Eldrup *et al.*¹¹ for vacancy migration and void formation in annealing stage III.

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